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Automotive fuels - Fatty acid methyl esters (FAME) for diesel engines - Requirements and test methods

Carburants pour automobiles - Esters méthyliques d'acide gras (EMAG) pour moteurs diesel - Exigences et méthodes d'essais

Kraftstoffe für Kraftfahrzeuge - Fettsäure-Methylester (FAME) für Dieselmotoren - Anforderungen und Prüfverfahren

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COMITÉ EUROPÉEN DE NORMALISATION
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Contents

Foreword

This document (prEN 14214:2002) has been prepared by Technical Committee CEN/TC 19 "Petroleum products, lubricants and related products", the secretariat of which is held by NEN.

This document is currently submitted to the Formal Vote.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

This European standard exists in parallel with EN 590 “Automotive fuels - Diesel - Requirements and test methods”.

This standard gives all relevant characteristics, requirements and test methods for FAME, which are known at time to be necessary to define the product to be used as automotive diesel fuel, including iodine value. The stability characteristics of FAME are under investigation in an EU-funded research programme ‘BIOSTAB’, and suitable limits and test methods may be incorporated into an amended version of this standard upon successful conclusion of this programme, including a possible replacement for iodine value.

Many of the test methods included in this standard were the subject of inter-laboratory testing to determine the applicability of the method and its precision in relation to different sources of fatty acid methyl esters. These fatty acid methyl esters were produced from rapeseed and sunflower oil.

Annex A is normative and contains the precision data generated on the test methods which are the result of the interlaboratory testing as mentioned above, carried out by working groups of CEN/TC 19. Annex B and C, also normative, contain details on two calculations.

1 Scope

This European Standard specifies requirements and test methods for marketed and delivered fatty acid methyl esters (FAME) to be used either as automotive fuel for diesel engines at 100% concentration, or as an extender for automotive fuel for diesel engines in accordance with the requirements of EN 590. At 100% concentration it is applicable to fuel for use in diesel engine vehicles designed or subsequently adapted to run on 100% FAME.

NOTE: For the purposes of this European Standard, the terms "% (*m/m*)" and "% (*V/V*)" are used to represent respectively the mass fraction and the volume fraction.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 116:1997, *Diesel and domestic heating fuels – Determination of cold filter plugging point*

EN 590:1997, *Automotive fuels - Diesel - Requirements and test methods*

EN 12662:1998, *Liquid petroleum products - Determination of contamination in middle distillates*

prEN 14103:2001*, Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) – Determination of ester and linolenic acid methyl ester contents*

prEN 14104:2001*, Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) - Determination of acid value*

prEN 14105:2001, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of free and total glycerol and mono-, di-, triglyceride contents - Reference method*

prEN 14106:2001, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of free glycerol content*

prEN 14107:2001*, Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of phosphorus content by inductively coupled plasma (ICP) emission spectrometry*

prEN 14108:2001*, Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) – Determination of sodium content by atomic absorption spectrometry*

prEN 14109:2001*, Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) – Determination of potassium content by atomic absorption spectrometry*

prEN 14110:2001*, Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) - Determination of methanol content*

prEN 14111:2001, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of iodine value*

prEN 14112:2001*, Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of oxidation stability (accelerated oxidation test)*

prEN 14538:2002, *Fat and oil derivatives – Fatty Acid Methyl Esters (FAME) – Determination of Ca and Mg content by optical emission spectral analysis with inductively coupled plasma (ICP OES)*

EN ISO 2160:1998, *Petroleum products - Corrosiveness to copper - Copper strip test (ISO 2160:1998)*

EN ISO 3104:3104, *Petroleum products - Transparent and opaque liquids - Determination of kinematic viscosity and calculation of dynamic viscosity (ISO 3104:1994. incl. ISO Tech. Cor. N° 1)*

EN ISO 3170:1998, *Petroleum liquids – Manual sampling (ISO 3170:1988/A1:1998)*

EN ISO 3171:1998, *Petroleum liquids – Automatic pipeline sampling (ISO 3171:1988)*

EN ISO 3675:1998, *Crude petroleum and liquid petroleum products - Laboratory determination of density - Hydrometer method (ISO 3675:1998)*

EN ISO 4259:1995, *Petroleum products - Determination and application of precision data in relation to methods of test (ISO 4259:1992, including Cor. 1:1993)*

EN ISO 5165:1998, *Petroleum products - Determination of the ignition quality of diesel fuels - Cetane engine method (ISO 5165:1998)*

EN ISO 10370:1995, *Petroleum products - Determination of carbon residue (micro method) (ISO 10370: 1993)*

EN ISO 12185:1996/C1:2001, *Crude petroleum and petroleum products - Determination of density - Oscillating U-tube method (ISO 12185:1996, incl. ISO Tech. Cor. N° 1)*

EN ISO 12937:2000, *Petroleum products - Determination of water - Coulometric Karl Fisher titration method (ISO 12937:2000)*

EN ISO 13759:1996, *Petroleum products – Determination of alkyl nitrate in diesel fuels – Spectrometric method (ISO 13759:1996).*

prEN ISO 20846:2002, *Petroleum products – Determination of total sulfur content of liquid petroleum products – Ultraviolet fluorescence method (ISO/DIS 20846:2002)*

prEN ISO 20884:2002, *Petroleum products – Determination of low sulfur content of automotive fuels – Wavelength-dispersive X-ray fluorescence spectrometry (ISO/DIS 20884:2002)*

ISO/DIS 3679:3679*, Petroleum products - Determination of flash point - Rapid equilibrium closed cup method*

ISO 3987:1994*, Petroleum products - Lubricating oils and additives - Determination of sulfated ash*

ASTM D 1160:1999*, Distillation of Petroleum Products at Reduced Pressure*

3 Sampling

Samples shall be taken as described in EN ISO 3170 or EN ISO 3171 and/or in accordance with the requirements of national standards or regulations for the sampling of automotive diesel fuel. The national requirements shall be set out in a national annex to this European Standard, either in detail or by reference only.

In view of the sensitivity of some of the test methods referred to in this European Standard, particular attention shall be paid to compliance with any guidance on sampling containers, which is included in the test method standard.

4 Pump marking

Information to be marked on dispensing pumps used for delivering FAME diesel fuel, and the dimensions of the mark shall be in accordance with the requirements of national standards or regulations for the marking of pumps for automotive diesel fuel. Such requirements shall be set out in detail or shall be referred to by reference in a national annex to this European Standard.

5 Requirements and test methods

5.1 Dyes and markers

The use of dyes or markers is allowed.

5.2 Additives

In order to improve the performance quality, the use of additives is allowed. Suitable fuel additives without known harmful side effects are recommended in the appropriate amount, to help to avoid deterioration of driveability and emissions control durability. Other technical means with equivalent effect may also be used.

NOTE Deposit forming tendency test methods suitable for routine control purposes have not yet been identified and developed.

5.3 Generally applicable requirements and related test methods

5.3.1 When tested by the methods indicated in Table 1, fatty acid methyl esters (FAME) shall be in accordance with the limits specified in Table 1. The test methods listed in Table 1 have been shown to be applicable to fatty acid methyl esters in an inter-laboratory test programme. Precision data from this programme are given in normative Annex A, where these were found to be different from the precision data given in the test methods for petroleum products.

5.3.2 In case of a need for identification of FAME, a recommended method based on separation and characterisation of fatty acid methyl esters by LC/GC is prEN 14331 [1].

5.3.3 In case of a need for a check upon FAME quality, iodine value of FAME may be calculated by the method presented in Annex B (normative), but this method does not constitute an alternative to the iodine value requirement of Table 1.

5.3.4 The limiting value for the carbon residue given in Table 1 is based on product prior to addition of ignition improver, if used. If a value exceeding the limit is obtained on finished fuel in the market, EN ISO 13759 shall be used as an indicator of the presence of a nitrate-containing compound. If an ignition improver is thus proved present, the limit value for the carbon residue of the product under test cannot be applied. The use of additives does not exempt the manufacturer from meeting the requirement of maximum 0,30 % (*m/m*) of carbon residue prior to addition of additives.

Table 1 - Generally applicable requirements and test methods

Property	Unit	Limits		Test method [a]
		Minimum	Maximum	
Ester content [a]	% (*m/m*)	96,5 [b]		prEN 14103
Density at 15 °C [c]	kg/m^3	860	900	EN ISO 3675 EN ISO 12185
Viscosity at 40 °C [d]	mm^2/s	3,50	5,00	EN ISO 3104
Flash point	°C	120	–	ISO/DIS 3679 [e]
Sulfur content	mg/kg	–	10,0	prEN ISO 20846 prEN-ISO 20884
Carbon residue (on 10 % distillation residue) [f]	% (m/m)	–	0,30	EN ISO 10370
Cetane number [g]		51,0		EN ISO 5165
Sulfated ash content	% (m/m)	–	0,02	ISO 3987
Water content	mg/kg	–	500	EN ISO 12937
Total contamination [h]	mg/kg	–	24	EN 12662
Copper strip corrosion (3 h at 50 °C)	Rating	Class 1		EN ISO 2160
Oxidation stability, 110 °C	Hours	6,0	–	prEN 14112
Acid value	mg KOH/g		0,50	prEN 14104
Iodine value			120	prEN 14111
Linolenic acid methyl ester	% (m/m)		12,0	prEN 14103
Polyunsaturated (>= 4 double bonds) methyl esters [i]	% (m/m)		1	
Methanol content	% (m/m		0,20	prEN 14110
Monoglyceride content	% (m/m)		0,80	prEN 14105
Diglyceride content	% (m/m)		0,20	prEN 14105
Triglyceride content [j]	% (m/m)		0,20	prEN 14105
Free glycerol [j]	% (m/m)		0,02	prEN 14105 prEN 14106
Total glycerol	% (m/m)		0,25	prEN 14105
Group I metals (Na+K) [k] Group II metals (Ca+Mg) [l]	mg/kg mg/kg		5,0 5,0	prEN 14108 prEN 14109 prEN 14538
Phosphorus content	mg/kg		10,0	prEN 14107

[a] See 5.5.1

[b] The addition of non-FAME components other than additives is not allowed, see 5.2.

[c] Density may be measured by EN ISO 3675 over a range of temperatures from 20 ^{0}C to 60 ^{0}C. Temperature correction shall be made according to the formula given in Annex C. See also 5.5.2

[d] If CFPP is –20 °C or lower, the viscosity measured at -20 °C shall not exceed 48 mm^2/s. In this case, EN ISO 3104 is applicable without the precision data owing to non-Newtonian behaviour in a two-phase system.

[e] A 2 ml sample and apparatus equipped with a thermal detection device shall be used

[f] ASTM D 1160 shall be used to obtain the 10% distillation residue.

[g] See 5.5.3.

[h] See 5.5.1. An improved method is under development by CEN/TC 19.

[i] Suitable test method to be developed

[j] See also 5.5.1.

[k] Method under development. See Annex A for precision data for sum of Na + K

[l] See 5.5.1. Method under development. See Annex A for precision data for sum of Ca + Mg.

5.4 Climate dependent requirements and related test methods

5.4.1 For climate-dependent requirements options are given to allow for seasonal grades to be set nationally. The options are for temperate climates six CFPP (cold filter plugging point) grades and for arctic climates five different classes. Climate-dependent requirements are given in Table 2. Table 2 is divided into two sections, one for temperate climates (table 2a) and one for arctic climates (table 2b). When tested by the methods given in tables 2a and 2b, automotive diesel fuel shall be in accordance with the limits specified in these tables.

5.4.2 In a national annex to this European Standard each country shall detail requirements for a summer and a winter grade and may include (an) intermediate and/or regional grade(s) which shall be justified by national meteorological data.

Table 2 - Climate-related requirements and test methods

Table 2a - Temperate climates

Property	Unit	Limits						Test method [a]
		Grade A	Grade B	Grade C	Grade D	Grade E	Grade F	
CFPP	°C, max.	+5	0	-5	-10	-15	-20	EN 116
[a] See also 5.5.1.								

Table 2b - Arctic climates

Property	Units	Limits					Test method [a]
		class 0	Class 1	class 2	Class 3	class 4	
CFPP	°C, max.	-20	-26	-32	-38	-44	EN 116
[a] See also 5.5.1.							

5.5 Precision and dispute

5.5.1 All test methods referred to in this European Standard include a precision statement according to EN ISO 4259. In cases of dispute, the procedures described in EN ISO 4259 shall be used for resolving the dispute, and interpretation of the results based on the test method precision shall be used. However, the methods currently available for total contamination, ester content, triglyceride content, free glycerol and alkaline metals (Na + K) do not meet the 2R requirement of EN ISO 4259 at the limit in Table 1.

5.5.2 In cases of dispute concerning density, EN ISO 3675 shall be used with the determination carried out at 15 °C.

In cases of dispute concerning free glycerol, prEN 14105 shall be used.

5.5.3 For the determination of cetane number alternative methods may also be used in cases of dispute, provided that these methods originate from a recognized method series, and have a valid precision statement, derived in accordance with EN ISO 4259, which demonstrates precision at least equal to that of the referenced method. The test result, when using an alternative method, shall also have a demonstrable relationship to the result obtained when using the reference method.

Annex A
(normative)
Details of inter-laboratory test programme

Table of precision data from inter-laboratory test programme for requirements where precision differs from ISO/TC28 precision data

Property	Test method	Unit	CEN/TC19 data for pure FAME
Viscosity at 40 °C	EN ISO 3104	mm^2/s	r 0,11% R 1,8%
Flash point	ISO/DIS 3679	deg C	r 1,9 R 15,0
Sulfur content	prEN ISO 20884	mg/kg	r 0,026X + 1,356 R 0,0567X + 1,616
Cetane number	EN ISO 5165		r 2,4 R 5,0
Sulfated ash content	ISO 3987	%(m/m)	r $0,06X^{0,85}$ R $0,142X^{0,85}$
Total contamination	EN 12662	mg/kg	not available
CFPP	EN 116	deg C	not available
Distillation	ASTM D 1160	deg C	r 2,0 R 3,0 (90% distilled)

Precision data for the sum of Na + K, measured individually by prEN 14108 and prEN 14109, are as follows:

Repeatability r 0,017 * X + 0,512

Reproducibility R 0,305 * X + 1,980

Precision data for the sum of Ca + Mg, measured by prEN 14538, are as follows:

Repeatability r 0,0232X + 0,271

Reproducibility R 0,149X + 1,186

Annex B
(normative)
Calculation of Iodine Value

NOTE This method is adapted for biodiesel from the AOCS recommended practice Cd 1c – 85 for the determination of the iodine value of edible oil from its fatty acid composition [2].

B.1 Scope

This method describes a procedure for calculating the iodine value of neat biodiesel or biodiesel extracted from blends with diesel fuel.

B.2 Definition

This method is used to calculate the iodine value expressed in g I_2 /100 g sample from the percentage by mass of methyl esters as determined by either prEN 14103 (neat biodiesel) or prEN 14331 [1] (biodiesel extracted from blends with diesel fuel).

B.3 Procedure

The methyl ester composition of the sample is checked using the appropriate method as described in paragraph 2. The total methyl esters thus revealed should equal 100 after the deduction of the methyl ester C17 used for internal standard in prEN 14103.

The percentage by mass thus obtained is then used to calculate the sample's iodine value, being the sum of the individual contributions of each methyl ester, obtained by multiplying the methyl ester percentage by its respective factor (Table B.1), as indicated in the example in Table B.2.

The factor for each constituent of biodiesel is given in Table B.1.

Table B.1
METHYL ESTER FACTORS

Methyl ester	Factor
Methyl ester of saturated fatty acids	0
Methyl hexadecenoate (Methyl palmoleate) C16:1	0,950
Methyl octadecenoate (Methyl oleate) C18:1	0,860
Methyl octadecadienoate (Methyl lineolate) C18:2	1,732
Methyl octadecatrienoate (Methyl linolenate) C18:3	2,616
Methyl eicosenoate C20:1	0,785
Methyl docasenoate (Methyl erucate) C22:1	0,723

An example of the calculation of iodine value from the percentage by mass of methyl esters is given in Table B.2

Table B.2
CALCULATION EXAMPLE

Methyl ester of the following acids	Percentage % m/m	Factor	Contribution
Myristic C14:0	0,3	0	0
Palmitic C16:0	4,0	0	0
Palmitoleic C16:1	1,1	0,950	1,0
Stearic C18:0	2,0	0	0
Oleic C18:1	60,5	0,860	52,0
Linoleic C18:2	19,8	1,732	34,3
Linolenic C18:3	9,4	2,616	24,6
Eicosanoic C20:0	0,4	0	0
Eicosenoic C20:1	0,7	0,785	0,6
Docosanoic C22:0	0,7	0	0
Docosenoic C22:1	1,1	0,723	0,8
		Calculated I.V.	113,3

B.4 Expression of the result

Iodine value (calculated from the methyl ester composition) = X g I_2/100 g (B.1)

The result shall be reported to one decimal place.

NOTE 1 In 1994 the AOCS Uniform Methods Committee reviewed the coefficients used and concluded that no changes were necessary at that time. The present procedure uses the coefficients selected in the past for use in calculating the iodine value in triglyceride blends. The reasoning behind that choice is that triple the molecular weight of a methyl ester is almost identical to the molecular weight of the corresponding triglyceride.

NOTE 2 For samples with unsaponifiable content greater than 0,5% (m/m) or those containing a significant additive content, the calculated value tends to be higher than the true value.

NOTE 3 The calculated result tends to be lower than the true value in samples with a lower iodine value.

NOTE 4 In case of dispute the iodine value should be determined by prEN 14111.

Annex C
(normative)
Correction factor for calculation of density of FAME

The conversion factor for the correction of density, determined by EN ISO 3675 over a range of temperatures from 20 ^{0}C to 60 ^{0}C, to density at 15 ^{0}C is based on data published at the International Conference on Standardization and Analysis of Biodiesel, Vienna, November 1995 [3].

The density of seven samples of FAME was measured by pyknometer at 6 temperatures over the range 20 ^{0}C to 60 ^{0}C. The mean correction factor over the range was calculated as 0,723 kg/m^3.K, with a standard deviation of 1,2 % of this value. The average density of the FAME samples at 15 ^{0}C was calculated as 886,5 kg/m^3.

The following formula shall be used for the calculation of density of FAME at a certain temperature (*T*), determined by EN ISO 3675 over the range of temperatures from 20 ^{0}C to 60 ^{0}C:

$$\text{Density at } 15\ ^0\text{C, kg/m}^3 = \text{Density at } T\ ^0\text{C} + 0{,}723(T - 15) \qquad \text{(C.1)}$$

Bibliography

[1] prEN 14331, Liquid petroleum products – Separation and characterisation of fatty acid methyl esters (FAME) by liquid chromatography/gas chromatography (LC/GC).

[2] *The official Methods and Recommended Practices of the AOCS*, 5th edition, 1998, Champaign, IL, USA.

[3] J. Rathbauer & A. Bachler, *Physical Properties of Vegetable Oil Methyl Esters*, International Conference on Standardization and Analysis of Biodiesel, November 6th – 7th, 1995, Vienna.